

# **Iron Aluminide Hot Gas Filter Development**

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## **1.0 Introduction**

The development of advanced, coal fired, power generation systems such as pressurized fluid-bed combustion (PFBC) and integrated gasification combined cycles (IGCC) is an important part of the future energy picture for the United States and the world. These technologies can provide economical power generation with minimal environmental emissions and high efficiency. These advanced power generation projects are, however, dependent on the development of durable economical high temperature filter systems.

Currently high temperature filter systems are in the demonstration phase with the first commercial scale hot filter systems installed on IGCC units and demonstration units of PBFC systems. These filters are mostly ceramic tubes or candles. Ceramic filter durability has not been high. Failure is usually attributed to mechanical or thermal shock.

For IGCC the major problem associated with the use of ceramic filters is their lack of resistance to cracking due to mechanical loads. One possible solution to this problem is the development of sintered metal filters (which are more resistant to cracking than ceramic filters) which can withstand the hydrogen sulfide laden, high temperature gases of these systems. The purpose of this project is to develop crack resistant, corrosion resistant sintered metal filters of iron aluminide suitable for application in advanced power processes. The goal is to develop filters which will provide at least several years service in advance power gasification applications without a substantial temperature penalty.

## **2.0 Objective**

The overall objective of this project is to commercialize weldable, crack resistant metal filters which will provide several years service in advanced power generation processes. These filters will be used to remove particulates from the gas stream prior to entering a turbine.

The three objectives of the current portion of the project are to (1) develop filter media from corrosion resistant iron aluminide alloys, (2) develop manufacturing processes to make iron aluminide filters and (3) use a "short term" exposure apparatus supported by other tests to identify the most promising candidate (alloy plus sintering cycle). The objectives of the next phases are to demonstrate long term corrosion stability for the best candidate followed by the production of fifty filters (optional).

## **3.0 Approach**

Pall Corporation has taken the approach of developing sintered metal filter materials suitable for use in advanced power production applications. The focus of this project is to develop Iron aluminide for use in gasification environments. Specifically, three alloy compositions were chosen and modified after reviewing the relevant literature (1-7). The primary considerations for the alloys were resistance to spalling and corrosive attack in a reducing environment containing sulfur and chlorine and acceptable mechanical properties including ductility and tensile strength.

Powders produced by gas and water atomization techniques were reviewed. The powders were compared for the degree of green strength after compaction and by preliminary sinterability tests. The green strength will be needed during handling before the sintering operation and should increase the mechanical properties after the short sintering cycle.

The alloys chosen are: 0% Cr, 2% Cr and 5% Cr iron aluminide

## **4.0 Project Description**

This project is divided into three major tasks: The first task was to develop manufacturing methods for Iron Aluminide. This task was largely completed and reported on in our previous paper at the previous DOE conference. The second task was to assess the corrosion resistance of the iron aluminide in simulated gasification atmospheres. The third task (optional) is to produce 50 commercial scale filters.

This paper will focus on the second task.

### **4.1 Short Term Corrosion Testing**

Short-term corrosion testing of the three heat-treated iron aluminide compositions, plus one of the compositions in the non-oxidized state, was performed in simulated IGCC atmospheres (see Table I). These tests were used to identify the candidate alloy that has the best corrosion resistance combined with processing characteristics that will allow reliable manufacturing.

Table I

Representative IGCC Atmospheres and a Simulated Atmosphere for Exposure Testing

Types of Atmosphere	Oxygen Blown Tampa Electric	Air Blown Sierra Pacific	Simulated* Atmosphere (w/o Nitrogen) w/ chlorides
Temperature °F	900 - 925	1000 - 1050	Tampa - 925 Sierra - 1050 See Table II
Pressure	400 psia 26.1 atmosphere	272 - 275 psia	~ 1 atmosphere
<u>Component</u>	<u>Value - Mole %</u>	<u>Value - Mole %</u>	<u>Value - Mole %</u>
CO	40.36	28.89	37
H <sub>2</sub>	28.20	14.57	34
CO <sub>2</sub>	10.34	5.44	17
H <sub>2</sub> O	14.16	5.50	10
CH <sub>4</sub>	0.15	--	1.0
Ar	0.94	0.60	--
N <sub>2</sub>	5.13	48.65	--
COS	0.02	--	--
O <sub>2</sub>	0.00	0.00	0.00
H <sub>2</sub> S	0.63**	0.03**	Varied, See Table II
HCl	NA	NA	80 ppm
NaCl	NA	NA	2x*** 2 ppm
KCl	NA	NA	2x*** 5.5 ppm

\* Corresponds with oxygen blown Tampa Electric, Equilibrated at 1300°F, at 1 bar with no nitrogen.

\*\* Upstream of final desulfurization which is expected to lower H<sub>2</sub>S to 0.003% (30 ppm)

\*\*\* Amount added

**Note:** Temperatures and pressures supplied by METC.

#### **4.1.1 Heat-Treatment Testing**

The three filter compositions (2% chromium, 5% chromium, and 0% chromium grades) were heat-treated in circulated air at 800°C for 7 hours. The effect of heat treatment of the filters was tested with the 2% chromium grade only. There were two filters of this grade exposed during each run. One of the filters was heat-treated. The other filter was in the “as sintered” condition.

#### **4.1.2 Corrosion Test Apparatus**

A three zone, 11 kW, 4 inch diameter, 36 inch long solid tube furnace was used for the elevated temperature exposure testing. This furnace was linked to a second, 5.3 kW, 3.0 inch inner diameter, 24 inch tube furnace for preheating the atmosphere.

Both of the muffles for the furnaces were made of alonized stainless steel, a preferred containment material for atmospheres that have hydrogen sulfide as a constituent.

Both the furnaces were operated horizontally (see Figure 1). Temperature uniformity was favored by this positioning. The length of the uniform zone in the 4.0 inch diameter furnace was maximized to contain the four test filter elements. The tube that spanned the gap between the two furnaces containing the simulated atmosphere was insulated to reduce the loss of heat.

The four filter elements were attached end to end, with the final element blinded off, via the threaded hardware to make a “flow through” assembly. A graphite antisieze tape (Grafoil) was used on the NPT fittings to keep the individual test filters from galling and to make sure that the filter string could be disassembled after 1, 3, 7, and 14 days for non-destructive property testing. The string was then reassembled using the Grafoil tape. The filters were rotated in the filter string as is common practice in corrosion testing. A support was inserted between the second and third filters in the string to avoid creep during exposure.

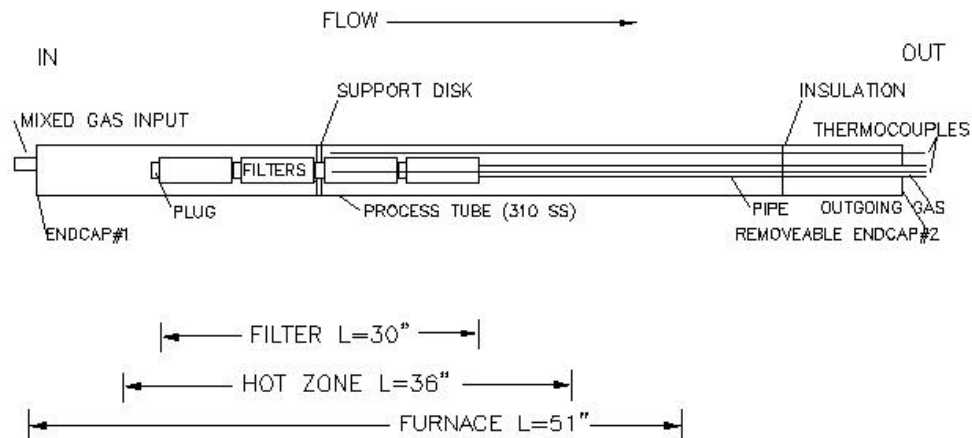
For temperature monitoring, two thermocouples were placed in the center of the hot zone length. One was inside the filter string while the other was on the outside of the filter string. The thermocouples were connected to a strip chart recorder providing a continuous record of temperature versus time.

#### **4.1.3 Blowback Testing**

Thermal pulsing was added to the exposure test to check the iron aluminide candidates for susceptibility to spalling the oxide scale. The following pulse parameters were chosen to simulate typical service conditions during blowback of filters:

- Pulse Duration = 0.75 s
- Pulse Frequency = every 15 min.
- Velocity = 18 ft/min.
- Pulse gas = Nitrogen
- Pulse Temperature = Room Temperature

The thermal pulsing was controlled by timed solenoid valves.



**Figure 1. Schematic of Process Tube Furnace for Short Term Exposure Testing**

#### **4.1.4 Test Atmosphere Components and Experimental Approach**

The atmospheres consisted of a mixture of hydrogen, carbon monoxide, methane, hydrogen sulfide and steam with sodium chloride, potassium chloride and hydrochloric acid. Table I lists the operating conditions for representative oxygen blown (Tampa Electric) and air blown (Sierra Pacific) IGCC atmospheres at system pressure. This table also lists the test atmosphere (without nitrogen) that was used, at approximately one atmosphere, to simulate both the oxygen blown and the air blown installations. The composition for this simulation atmosphere was determined by Oak Ridge National Laboratory (ORNL). The face velocity chosen was 0.5 feet per minute in forward flow. During the thermal pulsing the velocity was 18 feet per minute.

Hydrogen sulfide was dispensed from a tank (liquid phase). Provisions were made to measure the hydrogen sulfide levels before and after the gas passed through the filter string. Each day the H<sub>2</sub>S level was monitored at the inlet and at the outlet of the furnace tube. To measure the H<sub>2</sub>S levels a Toxic Gas Detector Model 8014KA (Matheson-Kitagawa) was used. The H<sub>2</sub>S inlet and outlet ports were hooked up in a tee, this allowed the gas to be flowing while the H<sub>2</sub>S was being measured. The hydrogen sulfide level outlet was kept within 15% of the target level.

Hydrogen, Carbon Monoxide, Carbon Dioxide and Methane were dispensed from individual pressurized cylinders.

A reservoir filled with DI water plus NaCl, KCl, and HCl supplied the water and chlorides to the test stand.

The furnace atmosphere flowed from the outside to the inside of the test filters which simulated use. The simulation gas was mixed in the process tube, flowed through the filters and then exited the furnace. Each run exposed four samples at one time.

Table II shows the matrix of seven test runs that were planned. Hydrogen sulfide and temperature were the principle variables. The proportions of the other atmosphere constituents were held constant for each hydrogen sulfide level.

#### **4.2 Material and Property Analysis**

Each of the exposed samples were analyzed and compared to the results of unused materials. The analyses completed on each sample are shown below:

##### **Material Analyses**

Carbon/Sulfur  
Chromium  
Tensile Test  
Ductility  
Metallographic examination  
SEM/EDX

##### **Property analyses**



Mass  
Air DeltaP  
Bubble Points

The air delta P was measured in inches of water across the filter media at a flow of 28 acfm/ft<sup>2</sup>.

For bubble point tests The samples were wet in and submerged approximately half inch below the surface of Filmex-B (denatured ethyl alcohol) prior to testing. Stoppers were placed in the open ends of the samples. Air pressure inside the element was gradually increased. The pressures at which the 1st and 10th bubbles occurred were recorded. The first bubble point is the pressure at which a bubble of air escapes from the largest pore in

**TABLE II**

Exposure Conditions with Hydrogen Sulfide and Temperature

	Gasifier	Higher Temperature			
					
		925°F		1050°F	1200°F
		Tampa Electric		Sierra Pacific	
 H <sub>2</sub> S	Variable (s)				
	H <sub>2</sub> S %	0.0783	0.0783	0.0783	0.0783
	Temp. °F	925	925	1050	1200
	Pulse	Y	N	Y	Y
	Chlorides	Y	N	Y	Y
	Run #	7	1	5	6
	Variable (s)				
	H <sub>2</sub> S %	0.783			0.783
	Temp. °F	925			1200
	Pulse	Y			Y
	Chlorides	Y			Y
	Run #	4			2
	Variable (s)				
	H <sub>2</sub> S %	7.83			
	Temp. °F	925			
	Pulse	Y			
	Chlorides	Y			
	Run #	3			

Comparison of Test Atmosphere an Actual Atmosphere

H<sub>2</sub>S Level Used for Short Term Exposure Test  
with Simulated Atmosphere at 1 atmosphere

0.0783 vol%  
0.783 vol%  
7.83 vol%

Equivalent H<sub>2</sub>S Level in Oxygen Blown  
Gasifier at 26.1 atmospheres

0.007 mol%  
0.072 mol%  
0.72 mol%

the sample: the first bubble point can be correlated to the absolute filter efficiency. The 10th bubble point was compared against the 1st bubble point to judge the uniformity of the pore size.

The open bubble point was also recorded. The open bubble point is an indication of the pressure required to pass a specified quantity of air (1 scfm/ft<sup>2</sup>) with the element wet in Filmex and relates by experience to the average pore size.

## **5.0 Results**

A summary of the results by alloy is presented in this section

### **5.1 0% Chromium Grade ( A Powder) Heat-treated**

The less desirable material properties and the relatively poor corrosion resistance of the 0% chromium grade powder eliminate it from further consideration. One of the 0% chromium grade test filters broke during the fourth exposure and was not replaced. The fracture occurred near the heat affected zone of the weld when it was located in the high stress region of the test string. (The filter attached to the tube that allows the gas to exit the test furnace.)

Increasing sintering temperature resulted in some improvement in ductility, however, it is not enough of an improvement to enable it to be considered for further use.

This composition also has poor weldability with the 310 stainless steel filler and 310 stainless steel hardware because of non-fusion. The weldability may be improved by using iron aluminide filler and solid iron aluminide end caps. This would increase the total cost of the final element and would also require development of reliable wrought material and machining parameters for the solid iron aluminide. The use of solid iron aluminide hardware should be reserved for the harshest exposure conditions and not as a possible solution to poor weldability.

### **5.2 5% Chromium Grade ( B Powder) Heat-treated**

The 5% chromium grade should have the benefit of better aqueous corrosion resistance than the 2% chromium grade. This can be very important during an unscheduled shut down of a filter system in application. Aqueous corrosion has not been tested during this experiment. The 5% chromium grade has a lower ductility than the 2% chromium grade, this may be able to be overcome with further optimization of forming, compressing and sintering parameters. The strength of this composition is consistently lower than the 2% chromium grade when manufactured under similar conditions.

The rate of weight gain of the 5% chromium grade was apparently linear. If the weight gain does not level out in longer tests this alloy should be eliminated from consideration. If a maximum weight gain is realized during longer term testing, and strength and



ductility improved, then the 5% chromium grade may be a better candidate for use in hot gas filter applications.

### **5.3 2% Chromium Grade ( C Powder) Heat-treated**

The heat-treated 2% chromium composition has had the best overall performance during the exposure tests. The combination of ductility and strength along with the small weight gains during the exposures make it the prime candidate for future evaluation.

Slight degradation of the material properties have occurred due to the exposure testing.

The current preferred composition, 2% chromium grade, was made by using half the standard value of Carbopol thickener, 37,000psi isostatic pressing, and 2420°F sintering. This produced an average ductility of 8.0%. A remarkable increase from the original 5.9% ductility at the beginning of task three. The increase in ductility has been accompanied by an increase in the strength. These process improvements result in a tougher, easier to weld product.

### **5.4 2% Chromium Grade ( C Powder) Non-Heat-treated**

The effect of heat-treatment was shown by comparing the weight gain results of the heat-treated to the as produced 2% chrome alloy. The non-heat-treated composition always showed a greater weight gain than the heat-treated composition.

The apparent parabolic weight gain of this grade in the third corrosion run is attributed to the corrosion and subsequent spalling of the 316L end caps. This spalling induced weight loss hides what was probably a weight gain for the medium.

### **5.5 Conclusions**

The heat-treated 2% chromium iron aluminide porous metal media is the preferred choice for IGCC, based on the combined strength, ductility, weldability, modulus of rupture and corrosion test results.

An effective, repeatable and scalable manufacturing process has been developed for three alloys of iron aluminide filtration media.

Iron aluminide filter materials manufactured utilizing the methods developed have physical/mechanical properties which are consistent with other porous metal media and are acceptable for use in IGCC.

The iron aluminide filter materials manufactured are capable of being fabricated, via existing cutting and welding methods, into filter elements suitable for commercial use.

The results of the short term corrosion tests conducted indicate that all the alloys manufactured into filter media have potential use in IGCC. These test results indicate that the heat-treated 2% chrome version has the highest chance for success.

There are indications that the manufactured heat-treated 5% chromium alloy version could be further optimized to produce equal or superior physical/mechanical properties to the heat-treated 2% chromium alloy.

There are indications that the corrosion resistance of 5% chromium version may be equal or better than the other alloys tested. This combined with the expected improved aqueous corrosion resistance indicate further testing is needed.

The manufacturing processes developed are not fully optimized and modifications which would improve carbon content, ductility, strength, corrosion resistance, manufacturability and costs are possible.

## **6.0 Application**

The iron aluminide media developed under this program has been specifically targeted for application in reducing environments which contain hydrogen sulfide and chlorides, i.e., coal gasification. The targets originally set were application at 1250 °F at H<sub>2</sub>S levels up to 3000 ppm<sub>v</sub>.

From the results of the tests to date, it appears that the material will provide long term operation at H<sub>2</sub>S levels substantially greater than those originally targeted.

This media was developed primarily for one reason. Our preliminary costing indicate that a full scale filter system utilizing iron aluminide media can have a capital cost 30% less than one designed exclusively for ceramic filter elements. This is a significant economic driver to pursue commercial application of iron aluminide filter elements in gasification systems.

In addition, with the sintering and manufacturing techniques developed in this program, we believe that it is possible to develop sintered metal filter elements which may be suitable for PFBC application. Initial discussions with ORNL indicate that 5% Cr iron aluminide may be suitable for application in PFBC. There are other alloys which are also promising which may be sinterable using the techniques developed.

## **7.0 Future Activities**

Pall has recently been given a contract extension to complete long term exposure testing of the candidate Iron aluminide media. This testing will take place this fall under simulated gasification conditions.

In addition, DOE has the option of directing Pall to manufacture 50 commercial scale iron aluminide filters for field testing in actual gasification operation.

## 8.0 Acknowledgments

The authors acknowledge the advice and encouragement of our METC Project Manager Ted McMahon. Special thanks are due to several individuals at Pall including Steve Geibel (PED) for guidance, to Joe Puzo (MMD) for help in the spinning and vacuum sintering of seamless cylinders and to Keith Rekczi (PED), a team member, for running experiments and for help in upgrading the exposure apparatus. Special thanks are due as well to several staff members at Oak Ridge National Laboratory; these include Peter Tortorelli and Jack de Van (Ret.) for calculating equilibrium atmospheres.

## 9.0 Contract Information

Research sponsored by the U. S. Department of Energy's Federal Energy Technology Center, under Contract DE-AC21-95MC31215 with Pall Corporation, 2200 Northern Blvd., East Hills, NY 11548, (516) 484-5400.

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